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(54) A method of treating a meat product to reduce a microbial population in the product

(57) Described is a method of sanitizing meat product using aqueous streams having an antimicrobial composition added to the stream. Preferably, the antimicrobial composition includes a mixture of one or more carboxylic acids having up to 18 carbon atoms and one or more peroxy carboxylic acids having up to 12 carbon atoms, preferably a mixture of a C<sub>2</sub>-C<sub>4</sub> peroxy carboxylic acid and a C<sub>8</sub>-C<sub>12</sub> peroxy carboxylic acid. Also described is a novel antimicrobial composition adapted for sanitizing meat product containing a mixture of one or more C<sub>2</sub>-C<sub>4</sub> peroxy carboxylic acids and one or more C<sub>8</sub>-C<sub>12</sub> peroxy carboxylic acids and an alpha-hydroxy mono- or dicarboxylic acid.

**Description****Field of the Invention**

5 [0001] This invention generally relates to compositions and processes for cleaning or sanitizing meat product during meat packing or preparation. More specifically, this invention relates to antimicrobial compositions and processes for cleaning and sanitizing meat product through direct contact between the meat product and the treatment. The nature of the contact between the meat product and the antimicrobial compositions improves antimicrobial properties. The 10 compositions and methods reduce microbial populations and do not affect the appearance, smell or taste of the meat product.

**Background of the Invention**

15 [0002] The cleaning of meat product in the preparation of any food product can be an unsanitary and time consuming task. Further, without a cleaning routine which follows an ordered process of steps to completely sanitize meat product, any number of problems may arise. Meat product may retain pathogens or infectious microbes (*E. coli*) or become increasingly more contaminated if viscera is allowed to rupture or is not properly removed. Further, incomplete cleaning of the meat product may also result in the presence of infectious microbes making the meat unsuitable for consumption.

20 [0003] PURAC® is a natural lactic acid produced by fermentation from sugar. It has a mild acid taste and is widely used in the food industry as an acidulant. PURAC® is an effective decontaminating agent for use with poultry, beef and pork carcasses and slaughter by-products. PURAC® is most effective at a use concentration of between 1 and 2 percent, and can be used at several different points in the slaughter line. Application immediately after hide removal reduces the amount of microorganisms entering subsequent processing steps, while treatments after evisceration and prior to chilling have the greatest residual effects. Mountney et al. also discuss the use of lactic acid to lower bacterial 25 counts and otherwise preserve poultry in "Acids As Poultry Meat Preservatives" in *Poultry Science*, 44: 582, 1965. Blankenship et al. discussed the destruction of *Salmonella* contaminates on fajita chicken meat in "Efficacy of Acid Treatment Plus Freezing To Destroy *Salmonella* Contaminates Of Spice Coated Chicken Fajita Meat" in *Poultry Science*, Vol. 69, Supp., 1990, p. 20. Adams et al. discuss the use of propylene glycol, sodium lactate, and lactic acid in chill water to reduce *salmonella* contamination of processed broilers. (See, Effects of Various Chill Water Treatments 30 on Incidents and Levels of *Salmonella* on Processed Carcasses, Department of Animal and Poultry Sciences, University of Arkansas, Fayetteville). Izat et al. discuss the effects of lactic acid on commercial broiler carcasses in reducing *salmonella* counts in *Poultry Science*, Vol. 69, Supp. 1990, p. 152; *Journal of Quality*, Vol. 13, 1990 p. 295-306; and *Journal of Food Protection*, Vol. 52, No. 9, pp. 670-673, Sept. 1989. Avens et al. discuss the pasteurization of turkey 35 carcasses and the reduction of *salmonella* using lactic acid in *Poultry Science*, Vol. 51, 1972, p. 1781. Mulder et al. in 1987 *Poultry Science* 66:1555-1557 reports a study of treating broiler carcasses with lactic acid, 1-cysteine and hydrogen peroxide. The treatment with lactic acid and hydrogen peroxide resulted in a 4-log cycle reduction in colony forming units of *Salmonella typhimurium*. Nevertheless, use of lactic acid resulted in a slightly changed color of the carcasses and all the treatments with hydrogen peroxide resulted in bleached and bloated carcasses.

40 [0004] Although peroxycarboxylic acids are known to be used for cleaning and sanitizing equipment and other surfaces, they have not been reported for cleaning and sanitizing meat product. Holzhauser et al., U.S. Patent 5,435,808, describes curing of animal hides with an acetic acid, peroxyacetic acid, hydrogen peroxide, and phosphoric acid combination. The heightened concerns of consumers over the organoleptic purity and safety of meat products, concerns over the environmental and organoleptic impact of many antimicrobial agents currently available, as well as the stringent economies of the meat and poultry industry have resulted in an ongoing need for meat product sanitizing compositions and processes which provide increased sanitization with organoleptic and environmental purity.

**Summary of the Invention**

50 [0005] Accordingly the present invention, in a first aspect, provides a method of treating meat product to obtain a reduction by at least one  $\log_{10}$  in surface microbial population which method includes the step of treating said meat product with an antimicrobial composition comprising an effective antimicrobial amount comprising at least 2 parts per million (ppm, parts by weight per each one million parts) of one or more peroxycarboxylic acids having up to 12 carbon atoms and an effective antimicrobial amount comprising at least 20 ppm of one or more carboxylic acids having up to 18 carbon atoms to reduce the microbial population.

55 [0006] A second aspect of the invention is an antimicrobial composition adapted for cleaning and sanitizing meat product which contains about 0.5 weight percent (wt-%) to about 20 wt-% of a mixture of one or more peroxycarboxylic acids having from 2-4 carbon atoms, and one or more peroxycarboxylic acids having from 8-12 carbon atoms, from about 0.5 wt-% to about 60 wt-% of an alpha-hydroxy mono or dicarboxylic acid having 3-6 carbon atoms, an effective

amount of a sequestrant and an effective amount of a hydrotrope.

[0007] A third preferred aspect of the present invention is an antimicrobial composition adapted for treating meat product consisting of a mixture of peroxyacetic and peroxyoctanoic acid in a ratio of about 10:1 to about 1:1, from about 0.1 to about 10 wt-% of lactic acid, from about 4 wt-% to about 10 wt-% of hydrogen peroxide and from about 0.5 wt-% to about 1.5 wt-% of a sequestering agent.

5 [0008] A fourth aspect of the present invention involves a method of treating a meat product to reduce a microbial population in the meat product, the method comprising the steps of spraying an aqueous antimicrobial treatment composition onto said meat product at a pressure of at least 50 psi at a temperature of up to about 60°C resulting in a contact time of at least 30 seconds, the antimicrobial composition comprising an effective antimicrobial amount comprising 10 least 2 ppm of one or more carboxylic acid, peroxyacrylic acid or mixtures thereof; and achieving at least a one  $\log_{10}$  reduction in the microbial population.

10 [0009] A fifth aspect of the present invention involves a method of treating a meat product to reduce a microbial population in the meat product, the method comprising the steps of placing the meat product in a chamber at atmospheric pressure; filling the chamber with condensing steam comprising an antimicrobial composition for a short duration; and 15 quickly venting and cooling the chamber to prevent browning of the meat product; wherein the duration of the steam thermal process may be from about 5 seconds to about 30 seconds and the chamber temperature may reach from about 50°C to about 93°C.

15 [0010] The antimicrobial composition can be applied in various ways to obtain intimate contact with each potential place of microbial contamination. For example, it can be sprayed on the meat product, or the meat product can be 20 immersed in the composition. Additional methods include applying a foamed composition and a thickened or gelled composition. Vacuum and or light treatments can be included, if desired, with the application of the antimicrobial composition. Thermal treatment can also be applied, either pre-, concurrent with or post application of the antimicrobial composition. We have found a preferred spray method for treating meat product with compositions of the invention involving spraying the meat product with an aqueous spray at a temperature less than about 60°C at a pressure of about 25 50 to 500 psi gauge wherein the spray comprises an effective antimicrobial amount of a carboxylic acid, an effective antimicrobial amount of a peroxyacrylic acid or mixtures thereof. These sprays can also contain an effective portion of a peroxy compound such as hydrogen peroxide and other ingredients such as sequestering agents, etc. We have found that the high pressure spray action of the aqueous treatment removes microbial populations by combining the 30 mechanical action of the spray with the chemical action of the antimicrobial materials to result in a surprisingly improved reduction of such populations on the surface of the meat product. All pressures are psig (or psi gauge). Differentiation of antimicrobial "cidal" or "-static" activity, the definitions which describe the degree of efficacy, and the official laboratory protocols for measuring this efficacy are important considerations for understanding the relevance of antimicrobial agents in compositions. Antimicrobial compositions may effect two kinds of microbial cell damages. The first is a truly 35 lethal, irreversible action resulting in complete microbial cell destruction or incapacitation. The second type of cell damage is reversible, such that if the organism is rendered free of the agent, it can again multiply. The former is termed bacteriocidal and the latter, bacteriostatic. A sanitizer and a disinfectant are, by definition, agents which provide antibacterial or bacteriocidal activity and achieve at least a five fold reduction (i.e., a five  $\log_{10}$  reduction) in microbial populations after a 30 second contact time (see AOAC method 960.09).

40 [0011] In contrast, a preservative is generally described as an inhibitor or bacteriostatic composition that simply retards growth in a reversible mode. For the purpose of this patent application, successful microbial reduction is achieved when the microbial populations are reduced by one  $\log_{10}$ . In this industry, the one  $\log_{10}$  microbial population reduction is the minimum acceptable for the processes. Any increased reduction in microbial population is an added benefit that provides higher levels of protection for processed meat product.

#### 45 Detailed Description of the Invention

[0012] The invention is a process for sanitizing meat product through treatment with aqueous streams containing an antimicrobial composition. The dip or spray methods used for meat product cleaning as well as sanitizing meat product generally include an effective antimicrobial concentration of one or more carboxylic acids and one or more peroxyacrylic acids.

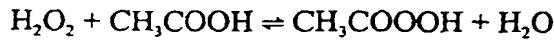
50 [0013] The term "meat product" encompasses all forms of animal flesh. The flesh of animals includes muscle, fat, organs, skin, bones and body fluids and like components that form the animal. Animal flesh includes the flesh of mammals, birds, fishes, reptiles, amphibians, snails, clams, crustaceans, or other edible species such as lobster, crab, etc. The forms of animal flesh include, for example, the whole or part of animal flesh, alone or in combination with other 55 ingredients. Typical forms include, for example, processed meats such as cured meats, sectioned and formed products, minced products, finely chopped products and whole products.

A. The Sanitizing Composition

[0014] The sanitizing composition used in the method of the invention generally contains one or more carboxylic acids and one or more peroxy carboxylic acids with a peroxygen compound such as  $H_2O_2$ . Typically, however, the composition 5 contains one or more carboxylic acids, an oxidizer, and one or more peroxy carboxylic acids depending on equilibrium. Commonly, the peroxy carboxylic acid material can be made by oxidizing a carboxylic acid directly to the peroxy carboxylic acid material which is then solubilized in the aqueous rinse agent compositions of the invention. Further, the materials can be made by combining the unoxidized acid with a peroxygen compound such as hydrogen peroxide to 10 generate the peracid in situ prior to blending the peroxy carboxylic acid with other constituents. The compositions of the invention comprises blends of the carboxylic acid and peroxy carboxylic acid along with other components including a peroxy source such as hydrogen peroxide. Once blended and applied the compositions can change due to interactions between the blended materials and due to interactions in the use locus. For example, the salt component can exchange 15 and become associated with free acids and the peroxy source can oxidize oxidizable materials. The anti-microbial properties arise from the blend of an acid material and a peracid material. The modification post blending and application do not change the invention.

[0015] A carboxylic acid is an organic acid ( $R-COOH$ ) which contains an aliphatic group and one or more carboxyl groups. A carboxyl group is represented by  $-COOH$ , and is usually located at a terminal end of the acid. The aliphatic group can be a substituted or unsubstituted group. Common aliphatic substituents include  $-OH$ ,  $-OR$ ,  $-NO_2$ , halogen, and other substituents common on these groups. An example of a simple carboxylic acid is acetic acid, which has the 20 formula  $CH_3COOH$ . A peroxy carboxylic acid is a carboxylic acid which has been oxidized to contain a terminal  $-COOOH$  group. The term peroxy acid is often used to represent a peroxy carboxylic acid. An example of a simple peroxy acid is peroxyacetic acid, which has the formula  $CH_3COOOH$ .

[0016] Generally when the peroxy carboxylic acid is formulated in accordance with the invention a monocarboxylic acid, such as acetic acid, is combined with an oxidizer such as hydrogen peroxide. The result of this combination is a 25 reaction producing a peroxy carboxylic acid, such as peroxyacetic acid, and water. The reaction follows an equilibrium in accordance with the following equation:



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wherein the  $pK_{eq}$  is 1.7.

[0017] The importance of the equilibrium results from the presence of hydrogen peroxide, the carboxylic acid and the peroxy carboxylic acid in the same composition at the same time. Because of this equilibrium, a mixture of carboxylic 35 acid and peroxy carboxylic acid can be combined in water without adding hydrogen peroxide. If permitted to approach equilibrium, the mixture will evolve hydrogen peroxide. This combination provides enhanced sanitizing with none of the deleterious environmental or organoleptic effects of other sanitizing agents, additives, or compositions.

THE CARBOXYLIC ACID

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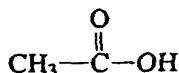
[0018] The first constituent of the composition used in the method of the invention includes one or more carboxylic acids. Generally, carboxylic acids have the formula  $R-COOH$  wherein the  $R$  may represent any number of different groups including aliphatic groups, alicyclic groups, aromatic groups, heterocyclic groups, all of which may be saturated or unsaturated. Carboxylic acids also occur having one, two, three, or more carboxyl groups. Aliphatic groups can be 45 further differentiated into three distinct classes of hydrocarbons. Alkanes (or paraffins) are saturated hydrocarbons. Alkenes (or olefins) are unsaturated hydrocarbons which contain one or more double bonds and alkynes (or acetylenes) are unsaturated hydrocarbons containing one or more highly reactive triple bonds. Alicyclic groups can be further differentiated into three distinct classes of cyclic hydrocarbons. Cycloparaffins are saturated cyclic hydrocarbons. Cycloolefins are unsaturated cyclic hydrocarbons which contain one or more double bonds while cycloacetylenes are 50 unsaturated cyclic hydrocarbons containing one or more highly reactive triple bonds. Aromatic groups are defined as possessing the unsaturated hydrocarbon ring structure representative of benzene. Heterocyclic groups are defined as 5 or 6 member ring structures wherein one or more of the ring atoms are not carbon. An example is pyridine, which is essentially a benzene ring with one carbon atom replaced with a nitrogen atom.

[0019] Carboxylic acids have a tendency to acidify aqueous compositions in which they are present as the hydrogen 55 atom of the carboxyl group is active and may appear as a cation. The carboxylic acid constituent within the present composition when combined with aqueous hydrogen peroxide generally functions as an antimicrobial agent as a result of the presence of the active hydrogen atom. Moreover, the carboxylic acid constituent within the invention maintains the composition at an acidic pH. The composition of the invention can utilize carboxylic acids containing as many as 18 car-

bon atoms. Examples of suitable carboxylic acids include formic, acetic, propionic, butanoic, pentanoic, hexanoic, heptanoic, octanoic, nonanoic, decanoic, undecanoic, dodecanoic, lactic, maleic, ascorbic, citric, hydroxyacetic, neopentanoic, neoheptanoic, neodecanoic, oxalic, malonic, succinic, glutaric, adipic, pimelic and subric acid.

[0020] Carboxylic acids which are generally useful are those having one or two carboxyl groups where the R group is a primary alkyl chain having a length of C<sub>2</sub> to C<sub>5</sub> and which are freely water soluble. The primary alkyl chain is that carbon chain of the molecule having the greatest length of carbon atoms and directly appending carboxyl functional groups. Especially useful are mono- and dihydroxy substituted carboxylic acids including alpha-hydroxy substituted carboxylic acid. A preferred carboxylic acid is acetic acid, which produces peroxyacetic acid to increase the sanitizing effectiveness of the materials. Acetic acid has the structure of the formula:

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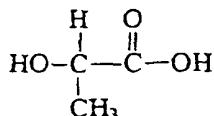


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[0021] An especially preferred  $\alpha$ -hydroxy-monocarboxylic acid is lactic acid, also known as 2-hydroxypropionic acid, which is a naturally occurring organic acid. Lactic acid has a molecular weight of 90.08 and is soluble in water, alcohol, acetone, ether and glycerol. Lactic acid occurs naturally and may be produced by fermentation. Alternatively, lactic acid may be synthesized.

Lactic acid has the structure of the formula

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The concentration of  $\alpha$ -hydroxy-mono- or di-carboxylic acid useful in the present invention generally ranges from about 0.5 wt-% to about 60 wt-%, preferably about 1 wt-% to about 20 wt-%, and most preferably from about 2 wt-% to about 10 wt-%. This concentration range of lactic acid is preferred for reasons of optimal acidity within the composition, as well as for the optimal antimicrobial efficacy which it brings to the antimicrobial system.

[0022] Reducing the concentration of lactic acid in comparison to any given concentration of hydrogen peroxide will essentially reduce the antimicrobial activity of the composition. Moreover, reducing the concentration of lactic acid may result in an increase in the pH of the composition and accordingly raise the potential for decreased antimicrobial activity. In sharp contrast, increasing the concentration of lactic acid within the present composition may tend to increase the antimicrobial activity of the composition. Furthermore, increasing the concentration of lactic acid in the composition of the present invention will tend to decrease the pH of the composition. Preferably, the pH of the present composition will be 4 or less with a generally preferred pH in the composition being between 1.5 and 3.75, and a pH between about 2 and 3.5 being most preferred.

[0023] Generally, the concentration of carboxylic acid within the composition used in the process of the invention ranges from about 0.5 wt-% to about 60 wt-%, preferably from about 10 wt-% to about 60 wt-%, and most preferably from about 20 wt-% to about 50 wt-%.

#### THE PEROXYCARBOXYLIC ACID

[0024] Another principle component of the antimicrobial composition of the invention is an oxidized carboxylic acid. This oxidized or peroxycarboxylic acid provides heightened antimicrobial efficacy when combined with hydrogen peroxide and the monocarboxylic acid in an equilibrium reaction mixture. Peroxycarboxylic acids generally have the formula R(CO<sub>3</sub>H)<sub>n</sub>, where R is an alkyl, arylalkyl, cycloalkyl, aromatic or heterocyclic group, and n is one or two and named by prefixing the parent acid with peroxy. An alkyl group is a paraffinic hydrocarbon group which is derived from an alkane by removing one hydrogen from the formula. The hydrocarbon group may be either linear or branched, having up to 12 carbon atoms. Simple examples include methyl (CH<sub>3</sub>) and ethyl (CH<sub>2</sub>CH<sub>3</sub>). An arylalkyl group contains both aliphatic and aromatic structures. A cycloalkyl group is defined as a cyclic alkyl group.

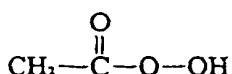
[0025] While peroxycarboxylic acids are not very stable, their stability generally increases with increasing molecular weight. Thermal decomposition of these acids may generally proceed by free radical and nonradical paths, by photo-

decomposition or radical-induced decomposition, or by the action of metal ions or complexes. Peroxycarboxylic acids may be made by the direct, acid catalyzed equilibrium action of 30-98 wt-% hydrogen peroxide with the carboxylic acid, by autoxidation of aldehydes, or from acid chlorides, acid anhydrides, or carboxylic anhydrides with hydrogen or sodium peroxide.

5 [0026] Peroxycarboxylic acids useful in this invention include peroxyformic, peroxyacetic, peroxypropionic, peroxybutanoic, peroxypentanoic, peroxyhexanoic, peroxyheptanoic, peroxyoctanoic, peroxynonanoic, peroxydecanoic, peroxyundecanoic, peroxydodecanoic, peroxylactic, peroymaleic, peroxyascorbic, peroxyhydroxyacetic, peroxyoxalic, peroxymalonic, peroxysuccinic, peroxyglutaric, peroxyadipic, peroypimelic and peroxysubric acid and mixtures thereof. These peroxycarboxylic acids have been found to provide good antimicrobial action with good stability in aqueous streams. In a preferred embodiment, the composition of the invention utilizes a combination of several different peroxycarboxylic acids. Preferably, the composition includes one or more small C<sub>2</sub>-C<sub>4</sub> peroxycarboxylic acids and one or more large C<sub>8</sub>-C<sub>12</sub> peroxycarboxylic acids. Especially preferred is an embodiment in which the small peroxycarboxylic acid is peroxyacetic acid and the large acid is either peroxyoctanoic acid or peroxydecanoic acid.

10 [0027] Peroxyacetic acid is a peroxycarboxylic acid with a structure as given the formula:

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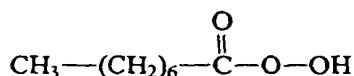


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wherein the peroxy group ,O-O- , is considered a high energy bond. Generally, peroxyacetic acid is a liquid having an acrid odor and is freely soluble in water, alcohol, ether, and sulfuric acid. Peroxyacetic acid may be prepared through any number of means known to those of skill in the art including preparation from acetaldehyde and oxygen in the presence of cobalt acetate. A 50% solution of peroxyacetic acid may be obtained by combining acetic anhydride, hydrogen peroxide and sulfuric acid. Other methods of formulation of peroxyacetic acid include those disclosed in U.S. Patent No. 2,833,813, which is incorporated herein by reference.

25 [0028] Peroxyoctanoic acid has the structure of the formula:

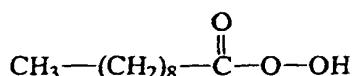
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[0029] Peroxydecanoic acid has the structure of the formula:

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45 [0030] The preferred peroxycarboxylic acid materials of the invention can be used to increase the sanitizing effectiveness of the materials. When a blended acid is used, the peroxycarboxylic acid is blended in proportions that range from about 10:1 to about 1:1 parts of C<sub>2</sub>-C<sub>4</sub> peroxycarboxylic acid per part of C<sub>8</sub>-C<sub>12</sub> peroxycarboxylic acid. Preferably, peroxyacetic acid is used at a ratio of about 8 parts per part of peroxyoctanoic acid.

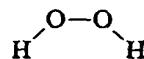
50 [0031] The above sanitizer material can provide antibacterial activity to the rinse aid sanitizers of the invention against a wide variety of microorganisms such as gram positive (for example, *Staphylococcus aureus*) and gram negative (for example, *Escherichia coli*) microorganisms, yeast, molds, bacterial spores, viruses, etc. When combined, the above peroxy acids can have enhanced activity compared to the low molecular weight peroxy acids alone.

55 [0032] Generally, the concentration of peroxycarboxylic acid within the composition used in the process of the invention ranges from about 0.5 wt-% to about 20 wt-%, preferably from about 2 wt-% to about 15 wt-%, and most preferably from about 4 wt-% to about 12 wt-%.

THE OXIDIZER

[0033] The composition used in the method of the invention also includes an oxidizer. Any number of oxidizers may be used as a precursor to the formation of a peroxycarboxylic acid as well as to provide further physical effervescent or agitation action to the composition of the invention. Preferably, the antimicrobial composition of the invention contains hydrogen peroxide. Hydrogen peroxide ( $H_2O_2$ ) has a molecular weight of 34.014 and it is a weakly acidic, clear, colorless liquid. The four atoms are covalently bonded in a non-polar structure:

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Generally, hydrogen peroxide has a melting point of -0.41°C, a boiling point of 150.2°C, a density at 25°C of 1.4425 grains per  $cm^3$ , and a viscosity of 1.245 centipoise at 20°C.

[0034] Hydrogen peroxide in combination with the carboxylic acid and peroxycarboxylic acid provides a surprising level of antimicrobial action against microorganisms, even in the presence of high loadings of organic sediment. Additionally, hydrogen peroxide provides an effervescent action which may irrigate any surface to which it is applied. Hydrogen peroxide works with a mechanical flushing action once applied which further plains the surface of application. An additional advantage of hydrogen peroxide is the food compatibility of this composition upon use and decomposition. For example, combinations of peroxyacetic acid and hydrogen peroxide result in acetic acid, water, and oxygen upon decomposition. All of these constituents are food product compatible. Generally, the concentration of hydrogen peroxide within the composition used in the process of the invention ranges from about 1 wt-% to about 35 wt-%, preferably from about 2 wt-% to about 25 wt-%, and most preferably from about 5 wt-% to about 10 wt-%. This concentration of hydrogen peroxide is most preferred as providing optimal antimicrobial effect.

[0035] These concentrations of hydrogen peroxide may be increased or decreased while still remaining within the scope of the present invention. For example, increasing the concentration of hydrogen peroxide may increase the antimicrobial efficacy of the claimed invention. Furthermore, increasing the hydrogen peroxide concentration may reduce the need to stabilize the hydrogen peroxide within the composition. Specifically, increasing the hydrogen peroxide concentration in the composition may provide a composition which has extended shelf life.

[0036] In contrast, decreasing the concentration of hydrogen peroxide may decrease the antimicrobial efficacy of the composition and necessitate the use of an increased concentration of carboxylic acid. Moreover, decreasing the concentration of hydrogen peroxide may necessitate the use of some stabilizing agent to ensure that the composition of the present invention will remain stable and efficacious over the intended time period.

[0037] In all, altering the concentration of the oxidizing agent will effect the equilibrium mix of the peroxycarboxylic acid used in the invention.

THE CARRIER

[0038] The composition of the invention also includes a carrier. The carrier functions to provide a reaction medium for the solubilization of constituents and the production of peroxycarboxylic acid as well as a medium for the development of an equilibrium mixture of oxidizer, peroxycarboxylic acid, and carboxylic acid. The carrier also functions to deliver and wet the antimicrobial composition of the invention to the intended substrate. To this end, the carrier may contain any component or components which will facilitate the functions. Generally, the carrier consists of water which is an excellent solubilizer and medium for reaction and equilibrium. The carrier may also include any number of constituents such as various organic compounds which facilitate the functions provided above. Organic solvents which have been found useful include simple alkyl alcohols such as ethanol, isopropanol, n-propanol, and the like. Polyols are also useful carriers in accordance with the invention, including propylene glycol, polyethyleneglycol, glycerol, sorbitol, and the like. Any of these compounds may be used singly or in combination with other organic or inorganic constituents or, in combination with water or in mixtures thereof. Preferably, the carrier consists of from about 1 wt-% to about 60 wt-% of an organic solvent.

[0039] Generally, the carrier makes up a large portion of the composition of the invention and may essentially be the balance of the composition apart from the active antimicrobial composition adjuvants, and the like. Here again, the carrier concentration and type will depend upon the nature of the composition as a whole, the environmental storage and method of application including concentration of the antimicrobial agent, among other factors. Notably the carrier should be chosen and used at a concentration which does not inhibit the antimicrobial efficacy of the act in the composition of the invention.

B. Adjuvants

[0040] The composition of the invention may also optionally include any number of adjuvants which are stable in an oxidizing environment, and add beneficial properties of stability, sequestration, sheeting and rinsing, etc. These adjuvants 5 may be preformulated with the sanitizing agent of the invention or added to the system simultaneously, or even after, the addition of the sanitizing agent of the invention.

Chelating Agent

[0041] The sanitizing agents of the invention may also contain a polyvalent metal complexing or chelating agent that 10 aids in reducing the harmful effects of hardness components and service water and improves product stability. The typically harmful effects of calcium, magnesium, iron, manganese, etc., ions present in service water can interfere with the action of either the washing compositions or rinsing compositions or can tend to decompose the active peroxxygen sanitizing materials. The chelating agent or sequestering agent can effectively complex and remove such ions from inappropriate interaction with active ingredients thus increasing sanitizing agent performance.

[0042] Both organic and inorganic chelating agents may be used. Inorganic chelating agents include such compounds 15 as sodium tripolyphosphate and other higher linear and cyclic polyphosphate species. Organic chelating agents include bath polymeric and small molecule chelating agents. Polymeric chelating agents commonly comprise polyanionic compositions such as polyacrylic acid compounds. Amino phosphates and phosphonates are also suitable for use as chelating agents in the compositions of the invention and include ethylene diamine (tetramethylene phosphonates), 20 nitrilotrismethylene phosphates, diethylenetriamine (pentamethylene phosphonates). These amino phosphonates commonly contain alkyl or alkaline groups with less than 8 carbon atoms.

[0043] Preferred chelating agents for use in this invention include improved food additive chelating agents such as 25 disodium salts of ethylene diamine tetraacetic acid or the well known phosphonates sold in the form of DEQUEST® materials, for example, 1-hydroxyethylidene-1,1-diphosphonic acid, etc. The phosphonic acid may also comprise a low molecular weight phosphonopolycarboxylic acid such as one having about 2-4 carboxylic acid moieties and about 1-3 phosphonic acid groups. Such acids include 1-phosphono-1-methylsuccinic acid, phosphonosuccinic acid and 2-phosphonobutane-1,2,4-tricarboxylic acid. Another organic phosphonic acid is (CH<sub>3</sub>C(PO<sub>3</sub>H<sub>2</sub>)<sub>2</sub>OH), available from 30 Monsanto Industrial Chemicals Co., St. Louis, MO, as DEQUEST® 2010, (which is a 58-62% aqueous solution; amino (tri(methylenephosphonic acid)] (N[CH<sub>2</sub>PO<sub>3</sub>H<sub>2</sub>]<sub>3</sub>), available from Monsanto as DEQUEST® 2000, as a 50% aqueous solution; ethylenediamine [tetra(methylenephosphonic acid)] available from Monsanto as DEQUEST® 2041, as a 90% solid acid product; and 2-phosphonobutane-1,2,4-tricarboxylic acid available from Mobay Chemical Corporation, Inorganic Chemicals Division, Pittsburgh, PA, as Bayhibit AM, as a 45-50% aqueous solution.

[0044] The above-mentioned phosphonic acids can also be used in the form of water soluble acid salts, particularly 35 the alkali metal salts, such as sodium or potassium; the ammonium salts or the alkylol amine salts where the alkylol has 2 to 3 carbon atoms, such as mono-, di-, or triethanolamine salts. If desired, mixtures of the individual phosphonic acids or their acid salts can also be used.

[0045] The concentration of chelating agent useful in the present invention generally ranges from about 0.01 to about 40 10 wt-%, preferably from about 0.1 to about 5 wt-%, most preferably from about 0.5 to about 2 wt-%.

Hydrotr pe

[0046] The sanitizing agent of the invention may also include a hydrotrope coupler or solubilizer. Such materials can 45 be used to ensure that the composition remains phase stable and in a single highly active aqueous form. Such hydrotrope solubilizers or couplers can be used at compositions which maintain phase stability but do not result in unwanted compositional interaction.

[0047] Representative classes of hydrotrope solubilizers or coupling agents include an anionic surfactant such as an alkyl sulfate, an alkyl or alkane sulfonate, a linear alkyl benzene or naphthalene sulfonate, a secondary alkane sulfonate, alkyl ether sulfate or sulfonate, an alkyl phosphate or phosphonate, dialkyl sulfosuccinic acid ester, sugar esters 50 (e.g., sorbitan esters) and a C<sub>8-10</sub> alkyl glucoside.

[0048] Preferred coupling agents for use in the rinse agents of the invention include n-octane sulfonate and aromatic sulfonates such as an alkyl benzene sulfonate (e.g., sodium xylene sulfonate or naphthalene sulfonate). Many hydrotrope solubilizers independently exhibit some degree of antimicrobial activity at low pH. Such action adds to the efficacy 55 of the invention but is not a primary criterion used in selecting an appropriate solubilizing agent. Since the presence of the peroxy carboxylic acid material in the proteinated neutral state provides beneficial biocidal or sanitizing activity, the coupling agent should be selected not for its independent antimicrobial activity but for its ability to provide effective single phase composition stability in the presence of substantially insoluble peroxy carboxylic acid materials and the more soluble compositions of the invention. Generally, any number of surfactants may be used consistent with the purpose

of this constituent.

[0049] Anionic surfactants useful with the invention include alkyl carboxylates, linear alkylbenzene sulfonates, paraffin sulfonates and secondary n-alkane sulfonates, sulfosuccinate esters and sulfated linear alcohols.

[0050] Zwitterionic or amphoteric surfactants useful with the invention include  $\beta$ -N-alkylaminopropionic acids, n-alkyl- $\beta$ -iminodipropionic acids, imidazoline carboxylates, n-alkyl- $\beta$ -lactaines, amine oxides, sulfobetaines and sulfaines.

[0051] Nonionic surfactants useful in the context of this invention are generally polyether (also known as polyalkylene oxide, polyoxyalkylene or polyalkylene glycol) compounds. More particularly, the polyether compounds are generally polyoxypropylene or polyoxyethylene glycol compounds. Typically, the surfactants useful in the context of this invention are synthetic organic polyoxypropylene (PO)-polyoxyethylene (EO) block copolymers. These surfactants have a diblock

10 polymer comprising an EO block and a PO block, a center block of polyoxypropylene units (PO), and having blocks of polyoxyethylene grafted onto the polyoxypropylene unit or a center block of EO with attached PO blocks. Further, this surfactant can have further blocks of either polyoxyethylene or polyoxypropylene in the molecule. The average molecular weight of useful surfactants ranges from about 1000 to about 40,000 and the weight percent content of ethylene oxide ranges from about 10-80% by weight.

15 [0052] Also useful in the context of this invention are surfactants including alcohol alkoxylates having EO, PO and BO blocks. Straight chain primary aliphatic alcohol alkoxylates can be particularly useful as sheeting agents. Such alkoxylates are also available from several sources including BASF Wyandotte where they are known as "Plurafac" surfactants. A particular group of alcohol alkoxylates found to be useful are those having the general formula  $R-(EO)_m-(PO)_n$  wherein m is an integer of about 2-10 and n is an integer from about 2-20. R can be any suitable radical such as

20 a straight chain alkyl group having from about 6-20 carbon atoms.

25 [0053] Other useful nonionic surfactants of the invention include capped aliphatic alcohol alkoxylates. These end caps include but are not limited to methyl, ethyl, propyl, butyl, benzyl and chlorine. Preferably, such surfactants have a molecular weight of about 400 to 10,000. Capping improves the compatibility between the nonionic and the oxidizers hydrogen peroxide and peroxycarboxylic acid, when formulated into a single composition. Other useful nonionic surfactants are alkyl/polyglycosides.

30 [0054] Another useful nonionic surfactant of the invention is a fatty acid alkoxylate wherein the surfactant comprises a fatty acid moiety with an ester group comprising a block of EO, a block of PO or a mixed block or heteric group. The molecular weights of such surfactants range from about 400 to about 10,000, a preferred surfactant has an EO content of about 30 to 50 wt-% and wherein the fatty acid moiety contains from about 8 to about 18 carbon atoms.

35 [0055] Similarly, alkyl phenol alkoxylates have also been found useful in the invention. Such surfactants can be made from an alkyl phenol moiety having an alkyl group with 4 to about 18 carbon atoms, can contain an ethylene oxide block, a propylene oxide block or a mixed ethylene oxide, propylene oxide block or heteric polymer moiety. Preferably such surfactants have a molecular weight of about 400 to about 10,000 and have from about 5 to about 20 units of ethylene oxide, propylene oxide or mixtures thereof.

40 [0056] The concentration of hydrotrope useful in the present invention generally ranges from about 0.1 to about 20 wt-%, preferably from about 0.5 to about 10 wt-%, most preferably from about 1 to about 4 wt-%.

#### Thickening/Gelling Agents

45 [0057] Thickeners useful in the present invention are those which do not leave contaminating residue on the surface of application, i.e., constituents which are incompatible with food or other sensitive products in contact areas.

50 [0058] Generally, thickeners which may be used in the present invention include natural gums such as xanthan gum. Also useful in the present invention are cellulosic polymers, such as carboxymethyl cellulose. Generally, the concentration of thickener use in the present invention will be dictated by the desired viscosity within the final composition. However, as a general guideline, viscosity of thickener within the present composition ranges from about 0.1 wt-% to about 1.5 wt-%, preferably from about 0.1 wt-% to about 1.0 wt-%, and most preferably from about 0.1 wt-% to about 0.5 wt-%.

#### C. Formulation

55 [0059] The compositions of the invention can be formulated by combining the sanitizing agent materials including other adjuvant components with the materials that form the sanitizer composition, the carboxylic acid or acid blend, hydrogen peroxide and optionally, hydrotrope solubilizer.

[0060] The compositions can also be formulated with preformed peroxycarboxylic acids. The preferred compositions of the invention can be made by mixing the carboxylic acid or mixture thereof with an optional hydrotrope solubilizer or 60 coupler, reacting the mixture with hydrogen peroxide and then adding the balance of required ingredients to provide rinsing and sanitizing action.

[0061] A stable equilibrium mixture is produced containing the carboxylic acid or blend with hydrogen peroxide and allowing the mixture to stand for 1-7 days at 15°C or more. With this preparatory method, an equilibrium mixture will be

formed containing an amount of hydrogen peroxide, unoxidized acid, oxidized or peroxy carboxylic acid and typically unmodified couplers, solubilizer, or stabilizers.

#### D. Use Compositions

5 [0062] The invention contemplates a concentrate composition which is diluted to a use solution prior to its utilization as a sanitizer. Primarily for reasons of economics, the concentrate would normally be marketed and an end user would preferably dilute the concentrate with water or an aqueous diluent to a use solution.

10 [0063] The general constituent concentrations of the sanitizing concentrate formulated in accordance with the invention may be found in Table 1:

Table 1

Constituent	Preferred (wt-%)	More Preferred (wt-%)	Most Preferred (wt-%)
H <sub>2</sub> O <sub>2</sub>	1-35	2-25	5-10
Peroxy carboxylic acids	0.5-20	2-15	4-12
Carboxylic acid	0.5-60	10-60	20-50
Chelating agent	0.01-10	0.01-5	0.5-2
Hydrotrope	0.1-20	0.5-10	1-4
Thickening agent	0.1-1.5	0.1-1.0	0.1-0.5
Carrier	0-97	10-90	12-65

15 [0064] The level of active components in the concentrate composition is dependent on the intended dilution factor and the desired activity of the peroxy carboxylic acid compound and the carboxylic acid.

20 [0065] Generally, a dilution of about 1 fluid ounce to about 0.5 to 10.0 gallons of water is used for aqueous antimicrobial sanitizing solutions. The composition shown in the preferred column of the Table 1 above would be used in a range from about 12.8 fluid ounce per gallon water to about 1 fluid ounce per 780 gallons of water depending on the desired level of peroxy carboxylic acid and concentration of the peroxy carboxylic acid in the product concentrate.

25 [0066] Higher use dilutions can be employed if elevated use temperature (greater than 25°C) or extended exposure time (greater than 30 seconds) can be employed. In the typical use locus, the concentrate is diluted with a major proportion of water and used for sanitizing using commonly available tap or service water mixing the materials at a dilution ratio of about 0.1 to about 2 ounces of concentrate per gallon of water.

30 [0067] Aqueous antimicrobial sanitizing use solutions can include at least about 2 ppm, preferably about 10 to about 500 ppm, and more preferably about 100 to about 250 parts per million of the peroxy carboxylic acid material; about 20 ppm to about 10,000 ppm, and preferably about 50 ppm to about 1,000 ppm of carboxylic acid; and about 10 to about 40 1,000 ppm of hydrogen peroxide. The aqueous use solution can further include at least about 50 ppm, preferably about 500 ppm of the hydrotrope solubilizer, and have a pH in the use solution in the range of about 1 to about 11 preferably about 2 to about 10.

#### E. Method of Use

45 [0068] During processing of the meat product, the meat product can be contacted with the compositions of the invention in any mode be that insures good contact between the meat product and the composition and at least some minimal mechanical work to result in at least a one log<sub>10</sub> reduction, preferably at least a two log<sub>10</sub> reduction and more preferably a three log<sub>10</sub> reduction in the resident microbial preparation. A five log<sub>10</sub> reduction in 30 seconds is a sanitizing treatment.

50 [0069] The invention is applicable to a wide range of meat product. For example, the antimicrobial compositions of the invention can be used on muscle meats, or any portion thereof, of any animal. The muscle meats most commonly consumed included, for example, beef, pork, veal, buffalo or lamb, sea food including scallops, shrimp, crab, octopus, mussels, squid or lobster and poultry including chicken, turkey, ostrich, game hen, squab or pheasant. The meat product may be in the form of whole, sectioned, processed, cooked or raw meats, such as, for example, hot dogs, cold cuts, sausage, meat cuts, hamburger, sushi and the like.

55 [0070] A preferred mode is a pressure spray with the sanitizing solution of the invention. During application of the spray solution on the meat product, the surface of the meat product can be moved with mechanical action, preferably

agitated, rubbed, brushed, etc. Agitation may be by physical scrubbing of the meat product, through the action of the spray solution under pressure or by other means. The agitation increases the efficacy of the spray solution in killing micro-organisms, perhaps due to better exposure of the solution into the crevasses or small colonies containing the micro-organisms. The spray solution, before application, may also be heated to a temperature of about 15 to 20°C, preferably about 20 to 50°C to increase efficacy. After a sufficient amount of time to kill the micro-organisms on the meat product, the spray solution may be rinsed off the meat product.

[0071] Application of the material by spray means can be accomplished using a manual spray wand application, an automatic spray of meat product moving along a production line using multiple spray heads to ensure complete contact or other spray means. One preferred automatic spray application involves the use of a spray booth. The spray booth substantially confines the sprayed composition to within the parameter of the booth. The production line moves the meat product through the entryway into the spray booth in which the meat product is sprayed on all its exterior surfaces with sprays within the booth. After a complete coverage of the material and drainage of the material from the meat product within the booth, the meat product can then exit the booth in a fully treated form. The spray booth can comprise steam jets that can be used to apply the antimicrobial compositions of the invention. These steam jets can be used in combination with cooling water to ensure that the treatment reaching the meat product surface is less than 65°C, preferably less than 60°C. The temperature of the spray on the meat product is important to ensure that the meat product is not substantially altered (cooked) by the temperature of the spray. The spray pattern can be virtually any useful spray pattern.

[0072] The spray can comprise a fogged material that leaves a fogging apparatus as a dispersion of fog particles in a continuous atmosphere. Such a spray has no defined pattern. The spray can have a pattern such as a conical spray in which the angle between the perimeter of the spray ranges from less than 180° to about 5°. Other spray patterns can also be useful. We have found that one preferred spray pattern involves a "fan" spray pattern in which the spray exits the spray head in a substantially planar form and the angle between the extent of the planar spray from edge to edge is about 20° or less, preferably about 15° or less. We have found that such a spray is preferred due to the increased mechanical action and efficiency of antimicrobial composition add on to the meat product. When such a narrow angle fan spray is used in a spray cabinet enclosure to treat the meat product, we have found that the optimum distance between the spray head and the meat product is less than about 100 centimeters, preferably about 20 to 80 centimeters, most preferably about 30 to 50 centimeters. Such a configuration efficiently transfers antimicrobial material to the meat product for efficient reduction of the microbial populations.

[0073] There are a number of parameters which need to be considered if spraying is the application method of choice. The first parameter to determine is the pressure at which the composition is sprayed onto the meat product. While spray pressures as low as about 25 psi (gauge) can be used with some valuable results, a higher spray pressure, greater than about 25, 50, 100, 150 psi and more preferably greater than about 200 psi, are effective in reducing the microbial populations due to the mechanical action of the spray on the meat product surface and on the microbial population remaining on the surface of the meat product. The spray action is best at temperatures less than 65°C. While a composition comprising lactic acid has been found to be most effective at low pressure, it has been discovered that equal, if not greater antimicrobial efficacy can be obtained by eliminating the lactic acid and merely increasing the spray application pressure. Further, if increased spray pressures are used, the antimicrobial composition can be applied at lower temperatures, potentially resulting in substantial energy savings. Of course there appears to be a relationship between application spray duration and antimicrobial efficacy. While spray durations of as little as about 10 seconds can be used, it has been discovered that a preferred spray duration is from about 10 to about 30 seconds. Without wishing to be limited by theory, the increased antimicrobial efficacy resulting from the use of the higher spray pressures is believed to be due to an improvement in penetrating the surface of the meat product, particularly an increased ability to reach into creases and crevices on the surface of the meat product.

[0074] During processing of the meat product, the meat product may also be immersed into a tank containing a quantity of sanitizing solution. The sanitizing solution is preferably agitated to increase the efficacy of the solution and the speed in which the solution kills micro-organisms attached to the meat product. Agitation can be obtained through conventional means including through ultrasonic means, aeration by bubbling air through the solution or by mechanical means, such as strainers, paddles, brushes, or pump driven liquid jets. The sanitizing solution may also be heated to increase the efficacy of the solution in killing micro-organisms. It is preferable that the meat product be immersed in the sanitizing solution after the meat product have been eviscerated and before any cooling process such as a chiller tank or a chill water spray.

[0075] In another alternative embodiment of the present invention, the meat product may be treated with a foaming version of the composition. The foam may be prepared by mixing foaming surfactants with the sanitizing solution at time of use. The foaming surfactants could be nonionic, anionic or cationic in nature. Examples of useful surfactant types include, but not limited to the following: alcohol ethoxylates, alcohol ethoxylate carboxylate, amine oxides, alkyl sulfates, alkyl ether sulfate, sulfonates, quaternary ammonium compounds, alkyl sarcosines, betaines and alkyl amides. The foaming surfactant is mixed at time of use with the sanitizing solution. Use solution levels of the foaming agents is from

about 50 ppm to about 2.0 wt-%. At time of use, compressed air is injected into the mixture, then applied to the meat product surface through a foam application device such as a tank foamer or an aspirated wall mounted foamer.

[0076] In another alternative embodiment of the present invention, the meat product may be treated with a thickened or gelled version of the composition. In the thickened or gelled state the sanitizing solution remains in contact with the meat product surface for longer periods of time, thus increasing the antimicrobial efficacy. The thickened or gelled solution will also adhere to vertical surfaces. The composition or the sanitizing solution may be thickened or gelled using existing technologies such as: xantham gum, polymeric thickeners, cellulose thickeners or the like. Rod micelle forming systems such as amine oxides and anionic counter ions could also be used. The thickeners or gel forming agents can be used either in the concentrated product or mixing with the sanitizing solution, at time of use. Typical use levels of thickeners or gel agents range from about 100 ppm to about 10 wt-%.

[0077] In another alternative embodiment of the present invention, the meat product may be treated with an electrostatically charged spray of the sanitizing solution. The sanitizing solution can be spray applied as a charged droplets by using conventional electrostatic spray technologies including inductively charged methodologies. As charged droplets, the sanitizing solution will be attracted to opposite or differentially charged surfaces such as the surface of the meat product. As a result, more sanitizing solution will be applied to the meat product surface and less solution will miss the intended target, commonly called over-spray. The charged droplets will also provide an evenly distributed solution layer on the meat product surface. The charged droplet size will range from about 10 microns to about 500 microns.

[0078] In another alternative embodiment of the present invention, the meat product may be subjected to a vacuum treatment either before applying the sanitizing solution, during the application of the sanitizing solution or after applying the sanitizing solution. When the meat product is subjected to a vacuum treatment in conjunction with the application of the sanitizing solution, the penetration of the sanitizing solution into the meat product substructure is enhanced. As a result, antimicrobial efficacy is improved. The amount of vacuum utilized is from about 2 inches of Mercury ("Hg) to about 29 inches of Mercury ("Hg).

[0079] In another alternative embodiment of the present invention, the meat product may be subjected to an activating light source following application of the sanitizing solution. The activating light can improve the antimicrobial efficacy of the sanitizing solution. The light source can be ultraviolet, infrared or from the visible spectrum.

[0080] The antimicrobial or sanitizing step can optionally be combined with a thermal intervention process which occurs either before, during or after the application of the antimicrobial composition. The thermal intervention process may employ hot water or dry heat. In the case of a hot water thermal process, the meat product is enclosed in a chamber at atmospheric pressure. The chamber is filled with condensing steam (finely divided liquid water) for a short duration, quickly vented, then cooled to prevent browning of the meat product. The duration of the steam thermal process may be from about 5 seconds to about 30 seconds. The chamber temperature may reach from about 50 °C to about 93°C. Similarly with dry heat, the meat product is placed in a chamber into which heated air is directed. The air is heated from about 65°C to about 260°C. The meat product is allowed from about 5 to about 30 seconds contact time with the heated air, the chamber is vented and the meat product is cooled.

#### Working Examples

[0081] The invention will now be described in more detail by reference to the following working examples. The only proper construction of these examples is as nonlimiting, illustrative example showing various formulations, stabilities, and applications of the invention.

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Test Formula #1	
Material	Weight Percent
Deionized water	53.9
Mixed Peroxycarboxylic acids <sup>1</sup>	4.75
Hydrogen Peroxide	6.9
Acetic Acid	25.0
Octanoic Acid	3.5
Hydroxyethylidene-1,1-diphosphonic acid	0.95
Sodium Octane mixed Mono- and Di-Sulfonate	5.0

1 A mixture of peroxy acetic and peroxyoctanoic acids is used. After application the composition is maintained in situ as a mixture of acid and peracid through the action of the H<sub>2</sub>O<sub>2</sub>.

Working Example #1

[0082] The objective of working example #1 was to determine if 0.5% and 1.0% lactic acid alone and in combination with Test Formula #1 and/or steam achieved a reduction in the bacterial flora present on prerigor beef samples. An exposure time of 10 minutes was utilized for all applications and testing was performed at 33°C.

**Operating Procedure:**

[0083] Sixteen prerigor beef samples were obtained and kept in a cooler until time of testing. Samples were aseptically divided in half. Eight different test treatments were utilized with four replicate pieces per treatment with the exception of the steam + 0.5% lactic acid treatment which only had three replicate pieces. Two cores (4.3 cm diameter) were taken from each replicate piece before and after treatment, combined into 99 mL of Phosphate Buffered Dilution water, stomached for 1 minutes and then serially diluted and plated using pour plate technique.

Test Products: 1. Test Formula #1 at 200 ppm Total Peracid  
 2. Test Formula #1 at 200 ppm Total Peracid + 0.5% Lactic Acid  
 3. Test Formula #1 at 200 ppm Total Peracid + 1.0% Lactic Acid  
 4. 0.5% Lactic Acid  
 5. Steam Alone, followed by a sterile water rinse  
 6. Steam + Test Formula #1 at 200 ppm Total Peracid, followed by a sterile water rinse  
 7. Steam + Test Formula #1 at 200 ppm Total Peracid + 0.5% Lactic Acid, followed by a sterile water rinse  
 8. Steam + 0.5% Lactic Acid followed by a sterile water rinse

Peracid Product Titrated	Actual Titrated
Peracid	
Test Formula #1 at 200 ppm	212 ppm
200 ppm Test Formula #1 + 0.5% Lactic Acid	220 ppm
200 ppm Test Formula #1 + 1.0% Lactic Acid	192 ppm
Test Formula #1 + Steam	210 ppm
Test Formula #1 + 0.5% Lactic Acid + Steam	220 ppm

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Product Application: All product use solutions were applied by a spray application for 10 seconds. This delivered approximately 150 mL of product. An exposure time of 10 minutes was utilized, followed by a 10 second sterile water rinse, if applicable.

5      Neutralizer:      99 mL of Phosphate Buffered Dilution Water

Dilutions Plated:       $10^0$ ,  $10^{-1}$ ,  $10^{-2}$  for Total Plate Count Before  
 $10^0$ ,  $10^{-1}$  for Total Plate Count After

10     Plating Medium:      Tryptone Glucose Extract Agar

Incubation:      26°C for 72 hours

15

Steam Application Parameters	
Test Product Application	Observations
Steam Alone	<p>1st Replicate: Starting temperature was 86°C, ending at 92°C. A 17 second exposure time was utilized and a 10 second delay occurred prior to the sterile water rinse for 10 seconds.</p> <p>2nd Replicate: Starting temperature was high 80°C, ending at 90+°C.</p> <p>3rd &amp; 4th Replicates: Starting temperature was 82°C, ending at 87°C. An 8 second exposure time and a 10 second sterile water rinse were utilized for replicates 2, 3 and 4.</p>
Test Formula #1 + Steam	<p>1st Replicate: Starting temperature was 82°C, ending at 87°C.</p> <p>2nd Replicate: Starting temperature was 80°C, ending at 84°C.</p> <p>3rd Replicate: Starting temperature was 83°C, ending at 88°C.</p> <p>4th Replicate: Starting temperature was 86°C, ending at 89°C.</p> <p>An 8 second exposure time and a 10 second sterile water rinse were utilized for all replicates.</p>
Test Formula #1 + 0.5% Lactic Acid + Steam	<p>1st Replicate: Starting temperature was 88°C, ending at 91.5°C.</p> <p>2nd Replicate: Starting temperature was 86.7°C, ending at 90+°C.</p> <p>3rd and 4th Replicates: Temperatures were not recorded.</p> <p>An 8 second exposure time and a 10 second sterile water rinse utilized for all replicates.</p>
0.5% Lactic Acid + Steam	<p>1st Replicate: Starting temperature was 84°C, ending at 88°C.</p> <p>2nd Replicate: Starting temperature was not recorded, however the ending temperature was 91°C.</p> <p>3rd Replicate: Temperatures were not recorded.</p> <p>An 8 second exposure time and a 10 second sterile water rinse were utilized for all replicates.</p>

55

Total Plate Count Results				
	Product Application	Average CFU/100 mL Before	Avg. CFU/100 mL After	Avg. $\log_{10}$ Reduc.
5	Test Formula #1 at 200 ppm	$3.3 \times 10^4$	$7.7 \times 10^3$	.80
10	Test Formula #1 at 200 ppm +0.5% Lactic Acid	$2.0 \times 10^5$	$1.7 \times 10^4$	1.08
15	Test Formula #1 at 200 ppm +1.0% Lactic Acid	$4.4 \times 10^4$	$1.2 \times 10^3$	1.31
20	0.5% Lactic Acid	$2.7 \times 10^4$	$5.4 \times 10^3$	0.91
25	Steam Alone with Sterile Water Rinse	$1.2 \times 10^4$	$2.4 \times 10^3$	1.10
	Steam + Test Formula #1 at 200 ppm with Sterile Water Rinse	$1.5 \times 10^4$	$8.4 \times 10^2$	1.51
	Steam + Test Formula #1 at 200 ppm + 0.5 % Lactic Acid With Sterile Water Rinse	$3.1 \times 10^5$	$2.6 \times 10^3$	2.55
	Steam +0.5% Lactic Acid with Sterile Water Rinse	$2.5 \times 10^4$	$9.3 \times 10^2$	1.69

**CONCLUSIONS:**

30 [0084] The application of Steam with Test Formula #1 at 200 ppm in combination with 0.5% Lactic Acid outperformed all other treatments by achieving an average of a  $2.55 \log_{10}$  reduction on the surface of prerigor meat. Steam alone, provided an average  $1.10 \log_{10}$  reduction with temperatures ranging from 80-92°C. Test Formula #1 at 200 ppm in combination with 0.5% Lactic Acid only provided an average  $1.10 \log_{10}$  reduction in comparison to an average  $1.31 \log_{10}$  reduction in combination with 1.0% Lactic Acid.

35 [0085] The purpose of the remaining working examples was to determine if the use of higher spray pressures, particularly those above 100 psi, would increase the antimicrobial efficacy of the compositions of the invention.

**Working Example #2**

40 [0086] The objective of the testing was to determine the efficacy of various antimicrobial treatments with extended spray and exposure times against the bacterial flora of prerigor beef.

**Test Method/Parameters:**

45 [0087] Prerigor beef samples were obtained and kept in a cooler at ambient temperature until time of testing. Ten different test treatments were utilized with four replicates per treatment. Two cores (4.3 cm diameter) were taken as each replicate from one piece for both before- and after- treatment samples and combined into 99 mL of Lethen Broth. The cores/neutralizer mixtures were stomached for 1 minute and then serially diluted and plated using pour plate technique.

50 **Test Products:** Test Formula #1\* at 200 ppm Peracid = 0.42% (4.2 mL were added to 995.8 mL tap water)  
 Test Formula #1\* at 500 ppm Peracid (10.5 mL were added to 989.5 mL tap water)  
 0.5% Lactic Acid  
 \* Test Formula #1, batch # Sil20972, was titrated at 4.76% total peracid.

55 **Application:** Eight cores (2 cores per replicate) were placed onto a clean and sanitary screen. The cores were sprayed with the appropriate test product utilizing a 10- or 30- second spray application time. For each replicate, two cores were removed after a 10-minute exposure time and placed into a stomacher bag containing 99 mL of neutralizer.

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Neutralizer: 99 mL Lethen Broth

Dilutions:  $10^0, 10^{-1}, 10^{-2}$  for Total Plate Count Before  
 $10^0, 10^{-1}$  for Total Plate Count After

5

Plating Medium: Tryptone Glucose Extract Agar

Incubation: 26°C for 72 hours

10 Calculations:

Average CFU/plate = (All eight counts from four replicates / 4)

15

Average CFU/plate x 100 = Average CFU/100 mL = Y

$$\text{Average } \frac{\text{CFU}}{\text{cm}^2} = \frac{Y}{2\pi r^2}$$

20

Dilution = 10, 100, or 1000

r = 2.15 cm

2 = # of cores

25

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5	Product Application	Average CFU/100 mL Before	Avg. CFU/100 mL After	Avg. Log <sub>10</sub> Reduc.
10	Water Control 98°F 25 psi pressure, 10 sec. spray	$3.6 \times 10^4$	$4.7 \times 10^4$	0.05
15	Water Control 120° 50 psi pressure, 30 sec. spray	$1.2 \times 10^5$	$1.9 \times 10^5$	-0.21
20	0.5% Lactic Acid 98°F 25 psi pressure, 10 sec spray	$1.6 \times 10^4$	$1.5 \times 10^4$	-0.01
25	0.5% Lactic Acid 120°F 25 psi pressure, 10 sec spray	$1.0 \times 10^5$	$8.4 \times 10^4$	0.07
30	Test Formula #1 at 200 ppm Peracid 90°F 50 psi pressure, 10 sec spray	$2.3 \times 10^4$	$8.7 \times 10^3$	0.41
35	Test Formula #1 at 200 ppm Peracid 120°F 50 psi pressure, 10 sec spray	$1.5 \times 10^5$	$1.6 \times 10^4$	0.97
40	Test Formula #1 at 200 ppm Peracid 120°F 50 psi pressure, 30 sec spray	$9.0 \times 10^4$	$3.9 \times 10^4$	0.37
45	Test Formula #1 at 200 ppm Peracid 120° 25 psi pressure, 30 sec spray	$6.5 \times 10^5$	$6.4 \times 10^5$ $1.9 \times 10^4$ *	0.01 1.65*
50	Test Formula #1 at 500 ppm Peracid 98°F 25 psi pressure, 30 sec. spray	$4.5 \times 10^4$	$5.3 \times 10^3$	0.93
	Test Formula #1 at 500 ppm Peracid 120°F 25 psi pressure, 10 sec. spray	$4.9 \times 10^4$	$1.1 \times 10^4$	0.67

\*Average and Log<sub>10</sub> Reduction not including replicate #3.

#### CONCLUSIONS:

55 [0088] Overall, the highest reductions in the bacteria flora on the surface of prerigor beef were seen with the following treatments:

- Test Formula #1 at 200 ppm total peracid at 50 psi pressure with a 10-second spray time at 120°F achieved an

average 0.97  $\log_{10}$  reduction.

- **Test Formula #1** at 500 ppm total peracid at 25 psi pressure with a 30-second spray at 98°F achieved an average 0.93  $\log_{10}$  reduction.

5 [0089] In regard to temperature, 120°F resulted in higher efficacy with **Test Formula #1** at 200 ppm total peracid at 50 psi pressure with a 10-second spray time, with a 0.97  $\log_{10}$  reduction versus a 0.41  $\log_{10}$  reduction at 98°F.

**Working Example #3**

10 [0090] The objective of the testing was to determine the efficacy of **Test Formula #1** at 200 ppm total peracid with a high pressure application spray at 100°F against the bacterial flora of prerigor beef.

**Test Method/Parameters:**

15 [0091] Prerigor beef samples were obtained and kept in a cooler at ambient temperature until time of testing. Four different test treatments were utilized with four replicates per treatment. Two cores (4.3 cm diameter) were taken as each replicate from one piece for both before and after treatment samples and combined into 99 mL of Lethen Broth. The cores/neutralizer mixtures were stomached for 1 minute and then serially diluted and plated using pour plate technique.

20 **Test Product:** **Test Formula #1** at 200 ppm total peracid (Batch # Sil20972, was titrated at 4.76% total peracid)

25 **Application:** Eight cores (2 cores per replicate) were aseptically removed from each sample before treatment. These were used for the before treatment samples. The remaining sample was placed onto a clean and sanitary screen. The sample was then sprayed with Vortex at approximately 200 ppm total peracid utilizing a 5-, 10- or 30- second spray application time. For each replicate, two cores were removed after a 10-minute exposure time and placed into a stomacher bag containing 99 mL of neutralizer.

30 **Neutralizer:** 99 mL Lethen Broth

**Dilutions:**  $10^0, 10^{-1}, 10^{-2}$  for Total Plate Count Before  
 $10^0, 10^{-1}$  for Total Plate Count After

35 **Plating Medium:** Tryptone Glucose Extract Agar

**Incubation:** 26°C for 72 hours

**Calculations:**

40 
$$\text{Average CFU/plate} = (\text{All eight counts from four replicates} / 4)$$

$$\text{Average CFU/plate} \times 100 = \text{Average CFU/100 mL} = Y$$

$$45 \text{Average} \frac{\text{CFU}}{\text{cm}^2} = \frac{Y}{2\pi r^2}$$

50 
$$\text{Dilution} = 10, 100, \text{ or } 1000$$
  

$$r = 2.15 \text{ cm}$$
  

$$2 = \# \text{ of cores}$$

55

5	Product Application	Average CFU/100 mL Before	Avg. CFU/100 mL After	Avg. Log <sub>10</sub> Reduc.
10	Water Control ~230 psi pressure, 30 sec. spray	$5.6 \times 10^5$	$2.7 \times 10^4$	1.31
15	Test Formula #1 at 200 ppm Peracid ~230 psi pressure, 10 sec spray	$\sim 1.9 \times 10^6$	$\sim 2.1 \times 10^5$	-0.96
20	Test Formula #1 at 200 ppm Peracid ~230 psi pressure, 5 sec spray	$\sim 2.8 \times 10^6$	$2.0 \times 10^5$	1.15
25	Test Formula #1 at 200 ppm Peracid ~230 psi pressure, 30 sec spray	$2.1 \times 10^6$	<100	>2.90

**CONCLUSIONS:**

[0092] Test Formula #1 at 200 ppm peracid with a 30-second exposure time utilizing a high-pressure spray of 230 psi at the nozzle with a distance of approximately 75 cm achieved the highest reduction with <3.4 CFU/cm<sup>2</sup> surviving after a 10-minute exposure time at ~110°F. Utilizing this procedure, a >2.90 log reduction was achieved.

**Working Example #4**

[0093] The objective of the testing was to determine the efficacy of Test Formula #1 at approximately 50, 100 and 200 ppm total peracid with a high pressure application spray at elevated temperatures in comparison to Lactic Acid against the bacterial flora of prerigor beef.

**Test Method Parameters:**

[0094] Prerigor beef samples were obtained and kept in a cooler at ambient temperature until time of testing. Four different test treatments were utilized with four replicates per treatment. Two cores (4.3 cm diameter) were taken as each replicate from one piece for both before- and after-treatment samples and combined into 99 mL of Lethen Broth. The cores/neutralizer mixtures were stomached for 1 minute and then serially diluted and plated using pour plate technique.

40 **Test Product:** Test Formula #1 at 50, 100 and 200 ppm total peracid  
Lactic Acid (88% concentrate)  
(Batch # Sil20972, was titrated at 4.76% total peracid)

45 **Application:** Eight cores (2 cores per replicate) were aseptically removed from each sample before treatment. These were used for the before-treatment samples. The remaining sample was placed onto a clean and sanitary screen. The sample was then sprayed with Test Formula #1 at approximately 50, 100 or 200 ppm total peracid utilizing a 20- or 30-second spray application time. 0.5% Lactic Acid utilized only the 30-second spray application time. For each replicate, two cores were removed after a 10-minute exposure time and placed into a stomach bag containing 99 mL of neutralizer.

50 **Neutralizer:** 99 mL Lethen Broth

**Dilutions:**  $10^0, 10^{-1}, 10^{-2}$  for Total Plate Count before  
 $10^0, 10^{-1}$  for Total Plate Count After

55 **Plating Medium:** Tryptone Glucose Extract Agar

**Incubation:** 16°C for 72 hours

## Calculations:

Average CFU/plate = (All eight counts from four replicates / 4)

5                   Average CFU/plate x 100 = Average CFU/100 mL = Y

$$\text{Average } \frac{\text{CFU}}{\text{cm}^2} = \frac{Y}{2\pi r^2}$$

10                   Dilution = 10, 100 or 10000  
 r = 2.15 cm  
 2 = # of cores

15

	Product Application	Average CFU/100 mL Before	Avg. CFU/100 mL After	Avg. $\log_{10}$ Reduc.
20	Test Formula #1 at 200 ppm Peracid ~230 psi pressure, 30 sec. spray	$3.7 \times 10^4$	<100	>2.58
25	Test Formula #1 at 200 ppm Peracid ~230 psi pressure 20 sec. spray	$3.1 \times 10^5$	$3.3 \times 10^3$	2.00
30	Test Formula #1 at 100 ppm Peracid ~230 psi pressure, 30 sec. spray	$1.3 \times 10^6$	$7.9 \times 10^3$	2.22
35	Test Formula #1 at 100 ppm Peracid ~230 psi pressure 20 sec. spray	$2.0 \times 10^5$	$4.3 \times 10^2$	2.66
40	Test Formula #1 at 50 ppm Peracid ~230 psi pressure, 30 sec spray	$3.1 \times 10^5$	$6.3 \times 10^3$	1.70
	Test Formula #1 at 200 ppm Peracid ~65 psi pressure 30 sec. spray	$2.1 \times 10^5$	$8.7 \times 10^4$	0.38
	~0.5% Lactic Acid ~230 psi pressure, 30 sec. spray	$8.7 \times 10^5$	$2.3 \times 10^4$	1.58

CONCLUSIONS:

[0095] Test Formula #1 at 200 ppm total peracid sprayed for 30 seconds at ~230 psi pressure achieved the highest reduction of bacteria present on the surface of prerigor meat with a >2.58  $\log_{10}$  reduction. Test Formula #1 at 200 ppm sprayed for 30 seconds at ~65 psi pressure only achieved an average 0.38  $\log_{10}$  reduction.

Working Example #5

[0096] The objective of the testing was to determine the efficacy of Test Formula #1 and Lactic Acid against *Listeria innocua* ATCC 33090 with a high-pressure application spray at elevated temperatures.

Test Method/Parameters:

[0097] Prerigor beef samples were obtained and kept in a cooler at ambient temperature until time of testing. Samples were cut into 13 cm pieces and 2.0 mL of the inoculum (see Test System Preparation below) was spread evenly over the entire surface of the sample. Inoculated samples were then left at room temperature (~23°C) for ≥ 15 minutes. Four replicate samples were taken (two cores per replicate) before treatment. After each spray treatment, a 10-minute exposure time was utilized, and taken four replicate samples were taken (two cores per replicate) and stomached for 1

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minute, serially diluted and plated using pour plate technique.

5 **Treatments:**

1. Test Formula #1 at 200 ppm total peracid with ~ psi pressure spray, 30 second spray time.
2. Test Formula #1 at 200 ppm total peracid with ~150 psi pressure spray, 30 second spray time.
3. Test Formula #1 at 200 ppm total peracid with ~100 psi pressure spray, 30 second spray time.
4. Water Control with ~220 psi pressure spray, 30-second spray time.
5. ~0.5% - 0.75% Lactic Acid with ~220 psi pressure spray, 30-second spray time.
6. Test Formula #1 at 100 ppm total peracid with ~220 psi pressure spray, 30 second spray time.
7. Test Formula #1 at 200 ppm total peracid with ~220 psi pressure spray, 15 second spray time.

10 **Tiration of the Lactic Acid solution used 12 drops of 1N Sodium Hydroxide for the indicator color change. In preliminary titrations of a 0.5% Lactic Acid solution, 7 drops of 1N Sodium Hydroxide were needed. Therefore, the sample was estimated to be at a concentration between 0.175% and 1.0% Lactic Acid.**

15 **Test Temperature:** ~120°F

Test System: *Listeria innocua* ATCC 33090

20 **Test System**

25 **Preparation:** 25 grams of sterilized cow feces was added into 50 grams of sterile phosphate buffered dilution water and stomached for 1 minute. 60.0 grams from this fecal slurry was transferred to a sterile stomachacher bag and 6.0 mL of an ~10<sup>8</sup> CFU/mL *Listeria innocua* 24-hour broth culture (grown in BHI broth at 37°C) was added and mixed. This inoculum was therefore estimated at 10<sup>7</sup> CFU/mL, which yielded approximately 10<sup>5</sup> CFU/cm<sup>2</sup>.

Exposure Time: 10 minutes

Neutralizer: 99 mL Lethen Broth

30 **Dilutions:** 10<sup>-4</sup>, 10<sup>-5</sup>, 10<sup>16</sup> (for Before Treatment, Inoculation Numbers samples)  
10<sup>0</sup>, 10<sup>-1</sup>, 10<sup>-2</sup> (After Treatment samples)

35 **Plating Medium:** *Listeria* Selective Agar

Incubation: 26° for 72 hours

Calculations:

40 Average CFU/plate = (All eight counts from four replicates / 4)

Average CFU/plate x 100 = Average CFU/100 mL = Y

45 
$$\text{Average } \frac{\text{CFU}}{\text{cm}^2} = \frac{Y}{2\pi r^2}$$

50 
$$r = 2.15 \text{ cm} \quad 2 = \# \text{ of cores}$$

55

Product Application	Avg. CFU/100 mL After	Avg. Log <sub>10</sub> Reduc.
Test Formula #1 at 200 ppm Peracid ~220 psi pressure 30 second spray*	1.6 x 10 <sup>5</sup>	1.97

(continued)

Product Application	Avg. CFU/100 mL After	Avg. $\log_{10}$ Reduc.
Test Formula #1 at 200 ppm Peracid ~150 psi pressure 30 second spray*	$5.1 \times 10^4$	2.45
Test Formula #1 at 200 ppm Peracid ~100 psi pressure 30 second spray*	$1.4 \times 10^5$	2.03
Water Control ~220 psi pressure 30 second spray	$4.9 \times 10^5$	1.48
Lactic Acid ~220 psi pressure 30 second spray	$3.5 \times 10^5$	1.63
Test Formula #1 at 100 ppm Peracid ~220 psi pressure 30 second spray*	$1.6 \times 10^5$	1.97
Test Formula #1 at 200 ppm Peracid ~100 psi pressure 15 second spray*	$2.2 \times 10^5$	1.83

#### CONCLUSIONS:

[0098] Treatment at 200 ppm peracid with an ~150 psi spray for 30 seconds achieved an average  $2.45 \log_{10}$  reduction of *Listeria innocua* ATCC 33090. Lactic Acid achieved a  $1.63 \log_{10}$  reduction of this organism which was only slightly higher than the water control which achieved an average  $1.48 \log_{10}$  reduction.

[0099] The above discussion, examples, and data illustrate our current understanding of the invention. However, since many variations of the invention can be made without departing from the spirit and scope of the invention, the invention resides in the claims hereinafter appended.

#### 40 Claims

1. A method of treating a meat product to reduce a microbial population in the meat product, the method comprising the steps of:

45 (a) treating said meat product with an antimicrobial composition comprising:

(i) an effective antimicrobial amount comprising at least 2 ppm of one or more mono- or di-peroxycarboxylic acids having up to 12 carbon atoms; and

50 (ii) an effective antimicrobial amount comprising at least 20 ppm of one or more carboxylic acids having up to 18 carbon atoms; and

(b) reducing the microbial population.

2. The method of claim 1 wherein the population reduction comprises at least one  $\log_{10}$  reduction in the microbial population.

55 3. The method of claim 1 wherein the population reduction comprises at least two  $\log_{10}$  reduction in the microbial population.

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4. The method of claim 1 wherein the population reduction comprises at least three  $\log_{10}$  reduction in the microbial population.
5. The process of claim 2 wherein the population comprises a human pathogen.
6. The process of claim 4 wherein the population comprises *Escherichia coli*.
7. The method of claim 1 wherein the meat product is selected from a muscle meat including beef, pork, veal, buffalo or lamb.
10. The method of claim 1 wherein the meat product is sea food including scallops, shrimp, crab, octopus, mussels, squid or lobster.
15. The method of claim 1 wherein the meat product is poultry including chicken, turkey, ostrich, game hen, squab or pheasant.
20. 10. The method of claim 1 wherein the peroxycarboxylic acid comprises one or more peroxycarboxylic acids having from 2 to 4 carbon atoms and a peroxycarboxylic acid having from 8 to 12 carbon atoms.
25. 11. The method of claim 7 wherein the peroxycarboxylic acid comprises peroxyacetic acid and peroxyoctanoic or peroxydecanoic acid, or mixtures thereof.
12. The method of claim 10 wherein the peroxycarboxylic acid having from 2 to 4 carbon atoms is peroxyacetic acid and the peroxycarboxylic acid having from 8 to 12 carbon atoms is peroxyoctanoic acid resulting in a ratio of about 10 to about 1 parts by weight of peroxyacetic acid per each 1 part of carboxylic acid.
30. 13. The method of claim 1 wherein the carboxylic acid is acetic acid.
14. The method of claim 1 wherein the carboxylic acid is an alpha-hydroxy mono or dicarboxylic acid having from 3 to 6 carbon atoms.
35. 15. The method of claim 14 wherein the carboxylic acid is lactic acid.
16. The method of claim 1 wherein said antimicrobial composition comprises about 2 to 25 parts by weight of hydrogen peroxide per each one million parts of the composition.
40. 17. The method of claim 1 wherein said antimicrobial composition is applied to the meat product by means of a spray.
18. The method of claim 1 wherein said antimicrobial composition is applied to the meat product by means of a fog.
45. 19. The method of claim 1 wherein said antimicrobial composition is applied to the meat product by means of a foam.
20. The method of claim 1 wherein said antimicrobial composition is applied to the meat product by applying in the form of a thickened or gelled solution.
50. 21. The method of claim 1 wherein all or part of the meat product is dipped in said antimicrobial composition.
22. The method of claim 21 wherein a solution comprising the antimicrobial composition is agitated.
55. 23. The method of claim 1 which further includes a vacuum treatment step.
24. The method of claim 1 which further includes the step of applying an activated light source to said meat product.
25. An antimicrobial composition adapted for cleaning and sanitizing meat product comprising:
  - (a) about 0.5 wt-% to about 20 wt-% of a mixture of one or more peroxycarboxylic acids having from 2 to 4 carbon atoms and one or more peroxycarboxylic acids having from 8 to 12 carbon atoms;
  - (b) from about 0.5 wt-% to about 60 wt-% of an alpha-hydroxy mono or dicarboxylic acid having from 3 to 6 car-

bon atoms

- (c) an effective amount of a sequestrant; and
- (d) an effective amount of a hydrotrope.

5 26. The composition of claim 25 wherein the peroxycarboxylic acid is a mixture of peroxyacetic acid and peroxyoctanoic or peroxydecanoic acid.

10 27. The composition of claim 26 wherein the peroxycarboxylic acid is a mixture of peroxyacetic acid and peroxyoctanoic acid in a ratio of about 10:1 to about 1:1.

15 28. The composition of claim 25 wherein the alpha-hydroxy mono- or dicarboxylic acid is lactic acid.

20 29. The composition of claim 25 which further comprises about 1 wt-% to about 35 wt-% of hydrogen peroxide.

25 30. The composition of claim 25 which further comprises from about 0.01 wt-% to about 10 wt-% of a sequestering agent.

30 31. The composition of claim 30 wherein the sequestering agent is 1-hydroxyethylidene-1,1-diphosphonic acid.

35 32. The composition of claim 25 which further comprises from about 0.1 to about 20 wt-% of a hydrotrope.

40 33. The composition of claim 25 which further comprises from about 0.01 to about 10 wt-% of a thickening or gelling agent.

45 34. The composition of claim 25 which further comprises from about 1 to about 60 wt-% of an organic solvent.

50 35. An antimicrobial composition adapted for treating meat product consisting essentially of:

- (a) a mixture of peroxyacetic and peroxyoctanoic acid in a ratio of about 10:1 to about 1:1;
- (b) from about 0.1 wt-% to about 10 wt-% of lactic acid;
- (c) from about 4 wt-% to about 10 wt-% of hydrogen peroxide; and
- (d) from about 0.5 wt-% to about 1.5 wt-% of a sequestering agent.

55 36. The composition of claim 35 wherein the sequestering agent is 1-hydroxyethylidene-1,1-diphosphonic acid.

60 37. A method of treating a meat product to reduce a microbial population in the meat product, the method comprising the steps of:

- (a) spraying an aqueous antimicrobial treatment composition onto said meat product at a pressure of at least 50 psi at a temperature of up to about 60°C resulting in a contact time of at least 30 seconds, the antimicrobial composition comprising an effective antimicrobial amount comprising least 2 ppm of one or more carboxylic acid, peroxycarboxylic acid or mixtures thereof; and
- (b) achieving at least a one  $\log_{10}$  reduction in the microbial population.

65 38. The method of claim 37 wherein the antimicrobial composition comprises an effective antimicrobial amount comprising at least 2 ppm of one or more peroxycarboxylic acids having up to 12 carbon atoms; and at least 20 parts of one or more carboxylic acids having up to 18 carbon atoms.

70 39. The method of claim 37 wherein the peroxycarboxylic acid comprises peroxyacetic acid, peroxyoctanoic acid, peroxydecanoic acid or mixtures thereof.

75 40. The method of claim 37 wherein the carboxylic acid comprises acetic acid, lactic acid or mixtures thereof.

80 41. The method of claim 37 wherein the antimicrobial composition comprises at least about 5 wt-% hydrogen peroxide.

85 42. The method of claim 37 wherein the antimicrobial compositions are applied by means of an electrostatically accelerated spray.

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43. A method of treating a meat product to reduce a microbial population in the meat product, the method comprising the steps of:

5        placing the meat product in a chamber at atmospheric pressure;  
filling the chamber with condensing steam comprising an antimicrobial composition for a short duration; and  
quickly venting and cooling the chamber to prevent browning of the meat product; wherein the duration of the  
steam thermal process may be from about 5 seconds to about 30 seconds and the chamber temperature may  
reach from about 50 °C to about 93°C.

10      44. The method of claim 43 wherein the antimicrobial composition comprises an effective antimicrobial amount comprising at least 2 ppm of one or more peroxycarboxylic acids having up to 12 carbon atoms; and at least 20 parts of one or more carboxylic acids having up to 18 carbon atoms.

15      45. The method of claim 44 wherein the peroxycarboxylic acid comprises peroxyacetic acid, peroxyoctanoic acid, peroxydecanoic acid or mixtures thereof.

46. The method of claim 44 wherein the carboxylic acid comprises acetic acid, lactic acid or mixtures thereof.

47. The method of claim 44 wherein the antimicrobial composition comprises at least about 5 wt% hydrogen peroxide.

20      48. The method of claim 44 wherein the antimicrobial compositions are applied by means of an electrostatically accelerated spray.

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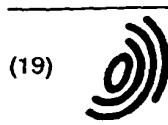
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### (54) A method of treating a meat product to reduce a microbial population in the product

(57) Described is a method of sanitizing meat product using aqueous streams having an antimicrobial composition added to the stream. Preferably, the antimicrobial composition includes a mixture of one or more carboxylic acids having up to 18 carbon atoms and one or more peroxycarboxylic acids having up to 12 carbon atoms, preferably a mixture of a C<sub>2</sub>-C<sub>4</sub> peroxycarboxylic acid and a C<sub>8</sub>-C<sub>12</sub> peroxycarboxylic acid. Also described is a novel antimicrobial composition adapted for sanitizing meat product containing a mixture of one or more C<sub>2</sub>-C<sub>4</sub> peroxycarboxylic acids and one or more C<sub>8</sub>-C<sub>12</sub> peroxycarboxylic acids and an alpha-hydroxy mono- or dicarboxylic acid.

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European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number

EP 99 11 6261

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
X	US 5 632 676 A (KURSCHNER ET AL.) 27 May 1997 (1997-05-27)	1,7-9, 13, 16-18,21	A23B4/12 A23B4/20 A23L3/3508
A	* column 2, line 28 - line 39 * * column 3, line 26 - line 42 * * column 3, line 49 - line 56 * * examples 1,4 *	11,25-36	A23L3/358 A23B4/14 A23B4/30
X	US 5 683 724 A (HEI ET AL.) 4 November 1997 (1997-11-04)	1,7, 9-13,21, 22	
A	* column 1, line 14 - line 19 * * column 2, line 5 - line 7 * * column 5, line 41 - line 51 * * column 5, line 52 - line 63 * * column 7, line 5 - line 8 * * examples 1,4,8 * * claims 4,5,9 *	35,36	
A	EP 0 233 731 A (INTEROX CHEMICALS) 26 August 1987 (1987-08-26) * page 3, line 9 - line 17 * * page 3, line 18 - line 29 * * page 4, line 22 - line 26 * * page 4, line 31 *	1,10,11, 25,26,35	TECHNICAL FIELDS SEARCHED (Int.Cl.7)
X	US 5 658 595 A (VAN OS) 19 August 1997 (1997-08-19) * column 3, line 45 - column 4, line 8 * * column 4, line 66 - column 5, line 8 * * column 5, line 22 - line 35 *	37,40,41	A23B A23L
A	WO 93 04595 A (KON-DES MILIEUTECHNOLOGIE) 18 March 1993 (1993-03-18) * page 2, line 32 - page 3, line 4 * * page 7, line 17 - line 29 *	37-42	
		-/-	
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
THE HAGUE	26 May 2000	Lepretre, F	
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone	T : theory or principle underlying the invention		
Y : particularly relevant if combined with another document of the same category	E : earlier patent document, but published on, or after the filing date		
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P : intermediate document	& : member of the same patent family, corresponding document		



DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
A	WO 97 38586 A (UNIVERSITY OF ARKANSAS) 23 October 1997 (1997-10-23) * example 6 *	37-42	
A	WO 83 03522 A (DOVER MEAT ADDITIVE COMPANY LTD) 27 October 1983 (1983-10-27) * page 5, line 1 - line 6; claims; figures *	37	
X	US 3 934 044 A (BUSCH ET AL.) 20 January 1976 (1976-01-20) * column 2, line 56 - column 4, line 32; examples *	43	
A	WO 96 13983 A (FRIGOSCANDIA INC.) 17 May 1996 (1996-05-17) * page 8, line 24 - page 9, line 35; figure 3 * * page 13, line 18 - line 29; figure 7 *	43-48	
A	EP 0 679 335 A (KENTMASTER MFG. CO. INC.) 2 November 1995 (1995-11-02) * column 5, line 41 - column 6, line 29 * * column 9, line 55 - column 12, line 12; figures *	43	TECHNICAL FIELDS SEARCHED (Int.Cl.7)
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
THE HAGUE		26 May 2000	Lepretre, F
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			
T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons S : member of the same patent family, corresponding document			



The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

1. Claims: 1-24 and 25-36

A method for treating a meat product to reduce a microbial population in the meat product comprising the steps of:  
 -treating the meat product with at least 2 ppm of a mono- or di-percarboxylic acid having up to 12 carbon atoms and at least 20 ppm of a carboxylic acid having up to 18 carbon atoms,  
 -reducing the microbial population

an antimicrobial composition comprising:  
 - a mixture of percarboxylic acids having from 2 to 4 carbon atoms and percarboxylic acids having from 8 to 12 carbon atoms,  
 - carboxylic acid having from 3 to 6 carbon atoms  
 -a sequestrant and a hydrotope.

and

an antimicrobial composition comprising:  
 -a mixture of peroxyacetic acid and peroxyoctanoic acid in a specific ratio,  
 -lactic acid,  
 -hydrogen peroxide  
 -a sequestering agent.

2. Claims: 37-42

A method of treating a meat product to reduce a microbial population comprising the steps of:  
 -spraying an aqueous antimicrobial composition comprising at least 2 ppm of one or more carboxylic acid, percarboxylic acid or mixtures thereof, at a pressure of at least 50 psi, a temperature of up to 60 C for a contact time of at least 30 seconds  
 -achieving at least a one log<sub>10</sub> reduction in the microbial population.

3. Claims: 43-48

A method of treating a meat product to reduce a microbial population comprising the steps of:  
 -contacting meat in a closed chamber with condensing steam comprising an antimicrobial composition for 5 to 30 seconds and at a chamber temperature of 50 C to 93 C,  
 -quickly venting and cooling the chamber.

**CLAIMS INCURRING FEES**

The present European patent application comprised at the time of filing more than ten claims.

Only part of the claims have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid, namely claim(s):

No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

**LACK OF UNITY OF INVENTION**

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

see sheet B

All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.

As all searchable claims could be searched without effort justifying an additional fee, the Search Division did not invite payment of any additional fee.

Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:

None of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:

ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.

EP 99 11 6261

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

26-05-2000

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 5632676	A	27-05-1997	AU	7800494 A	04-05-1995
			WO	9510191 A	20-04-1995
US 5683724	A	04-11-1997	US	5674538 A	07-10-1997
			US	5409713 A	25-04-1995
			AU	3758697 A	23-07-1998
			BR	9705133 A	25-05-1999
			CA	2214316 A	17-07-1998
			DE	19751391 A	23-07-1998
			FR	2758547 A	24-07-1998
			GB	2321242 A	22-07-1998
			IT	MI980016 A	17-07-1998
			JP	10210959 A	11-08-1998
			NZ	328744 A	19-12-1997
			AU	675975 B	27-02-1997
			AU	5088893 A	11-10-1994
			CA	2156299 A	29-09-1994
			CN	1092385 A	21-09-1994
			NZ	255871 A	27-08-1996
			WO	9421122 A	29-09-1994
EP 233731	A	26-08-1987	NONE		
US 5658595	A	19-08-1997	NL	9201178 A	01-02-1994
			NO	177481 B	19-06-1995
			AT	156669 T	15-08-1997
			AU	666335 B	08-02-1996
			AU	2646092 A	05-04-1993
			BR	9206477 A	31-10-1995
			CA	2118696 A	18-03-1993
			DE	69221628 D	18-09-1997
			DE	69221628 T	19-03-1998
			DK	603329 T	01-09-1997
			EP	0603329 A	29-06-1994
			ES	2106884 T	16-11-1997
			JP	7506239 T	13-07-1995
			WO	9304595 A	18-03-1993
			AU	2593792 A	05-04-1993
			WO	9304705 A	18-03-1993
WO 9304595	A	18-03-1993	NL	9201178 A	01-02-1994
			NO	177481 B	19-06-1995
			AT	156669 T	15-08-1997
			AU	666335 B	08-02-1996
			AU	2646092 A	05-04-1993
			BR	9206477 A	31-10-1995

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

EPO FORMS

ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.

EP 99 11 6261

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.  
The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

26-05-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9304595 A		CA 2118696 A DE 69221628 D DE 69221628 T DK 603329 T EP 0603329 A ES 2106884 T JP 7506239 T US 5658595 A AU 2593792 A WO 9304705 A	18-03-1993 18-09-1997 19-03-1998 01-09-1997 29-06-1994 16-11-1997 13-07-1995 19-08-1997 05-04-1993 18-03-1993
WO 9738586 A	23-10-1997	US 5855940 A AU 2672997 A CA 2250761 A EP 0912101 A US 6039992 A	05-01-1999 07-11-1997 23-10-1997 06-05-1999 21-03-2000
WO 8303522 A	27-10-1983	AU 1220683 A	04-11-1983
US 3934044 A	20-01-1976	CA 1039565 A	03-10-1978
WO 9613983 A	17-05-1996	AT 179303 T AU 696799 B AU 4104796 A BR 9509611 A CA 2203429 A CN 1171724 A DE 69509407 D DE 69509407 T EP 0790778 A ES 2135781 T JP 10508489 T US 5976005 A US 6019033 A US 5711981 A	15-05-1999 17-09-1998 31-05-1996 21-10-1997 17-05-1996 28-01-1998 02-06-1999 25-11-1999 27-08-1997 01-11-1999 25-08-1998 02-11-1999 01-02-2000 27-01-1998
EP 679335 A	02-11-1995	US 5503594 A US 5607349 A AT 176849 T AU 691254 B AU 1769695 A BR 9501822 A CA 2148027 A DE 69507910 D DE 69507910 T ES 2131232 T	02-04-1996 04-03-1997 15-03-1999 14-05-1998 09-11-1995 01-10-1996 29-10-1995 01-04-1999 23-09-1999 16-07-1999

ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.

EP 99 11 6261

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

26-05-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 679335 A		NZ 270963 A ZA 9503212 A	24-11-1997 03-01-1996